Final Report

on

Contract No. Nonr-495(04)
NR 055 298

Shalom Sarel and Melvin S. Newman
5 August 1954

THE OHIO STATE UNIVERSITY
RESEARCH FOUNDATION

RF Project 497
Report No. Final

FINAL

R E P O R T

By

THE OHIO STATE UNIVERSITY
RESEARCH FOUNDATION

Columbus 10, Ohio

To: OFFICE OF NAVAL RESEARCH
Contract No. Nonr-495(04)
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On: STERIC FACTORS IN ORGANIC CHEMISTRY

For the period: January 1, 1953 - July 31, 1954

Submitted by: Shalom Sarel and Melvin S. Newman
Department of Chemistry

Date: August 5, 1954

STERIC FACTORS IN ORGANIC CHEMISTRY

The object of this research program was to learn more about steric factors in the hydrolysis of esters of acetic acid. By comparing the rates of hydrolysis of a variety of primary alkyl acetates with that of ethyl acetate the steric effect of variations in the alkyl moiety can be evaluated if one assumes that substitutions on the β-carbon of the alkyl group have a negligible polar effect. In a similar way the steric effects in secondary alkyl acetates may be evaluated by comparing the rates of hydrolysis of secondary alkyl acetates with that of isopropyl acetate.

In this final report the synthesis of the alkyl acetates is described and the physical properties etc., of all compounds used in this research are tabulated.

The discussion of the significance of this work as well as the synthetic work involved is to be presented in two or three papers which are now being written for submission to the Journal of the American Chemical Society. Reprints of these papers will be mailed for distribution.

EXPERIMENTAL

Acetates of primary, secondary, and tertiary alcohols were prepared in good purity, and the rate of alkaline hydrolysis in 70% aqueous dioxane, or in water, at 20° and 30°C. was measured.

For the preparation of the primary and secondary alcohols and their acetates, the general methods used can be depicted as follows:

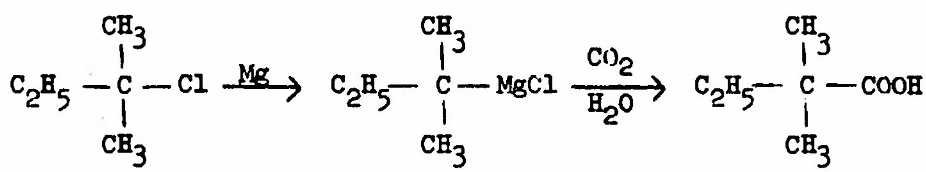
$$RCOOH \text{ or } RCOOR' \xrightarrow{LiAlH_4} RCH_2OH \xrightarrow{\text{acetylation}} RCH_2OCOCH_3$$

$$R-\overset{O}{\overset{\|}{C}}-R' \xrightarrow{LiAlH_4} RCHOHR' \xrightarrow{\text{acetylation}} R\overset{R'}{\overset{|}{C}}H-OCOCH_3$$

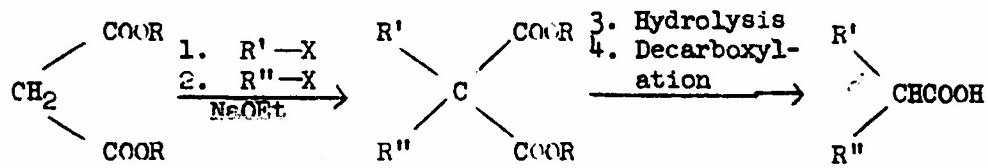
A. SYNTHESIS OF ACIDS

Six different methods were used for the preparation of the carboxylic acids or their esters. These methods are outlined below:

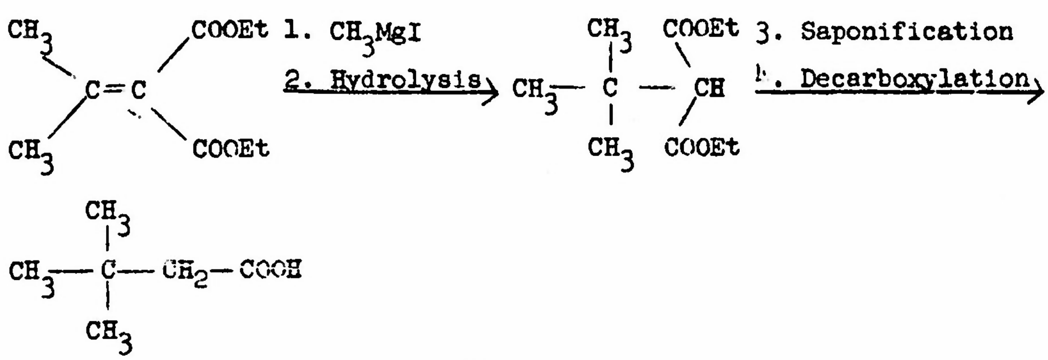
(1) Carboxylation of the Grignard Reagent:



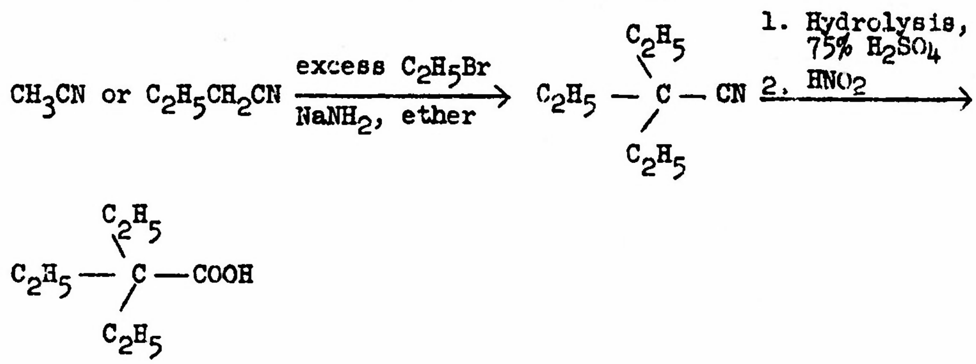
(2) Alkylation of Malonic or Cyanoacetic Esters:



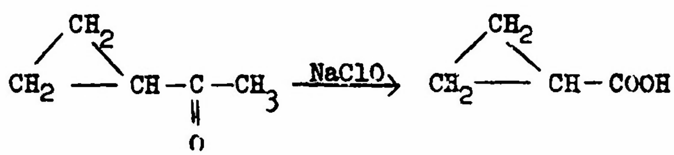
(3) 1.4-Addition of Grignard Reagent to Ethyl Alkylidene Malonate:



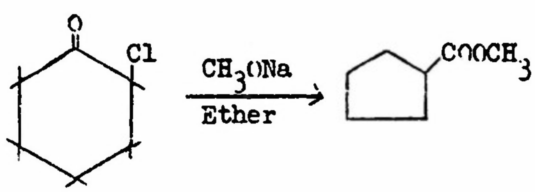
(4) Alkylation of Nitriles (Ziegler Method):

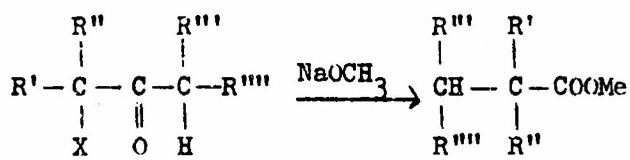


(5) Hypochlorite Oxidation of Methyl Ketones:



(6) Alkaline Rearrangement of α-Haloketones (Faworski Reaction):



In general: 

In method (6) the α-bromoketones were prepared either (a) by direct bromination of the corresponding ketones or (b) through replacement of hydroxyl group in an acyloin by bromine. The ketones themselves were prepared either by oxidation of an appropriate alcohol or through treatment of a cadmium dialkyl with the appropriate acyl halide.

B. FORMATION OF ALCOHOLS

Lithium Aluminum hydride was used as a reducing reagent for converting carboxylic acids and esters into the corresponding primary alcohols, and ketones to corresponding secondary alcohols.

C. FORMATION OF ACETATES

The new alcohols were smoothly and conveniently converted into their respective acetates by treatment with: (1) acetyl chloride or acetic anhydride and pyridine; or (2) isopropenyl acetate and acid; or (3) acetyl chloride and magnesium metal in dry ether as follows:

$$C_2H_5-\overset{\displaystyle C_2H_5}{\underset{\displaystyle C_2H_5}{\overset{|}{\underset{|}{C}}}}-OH + 2CH_3COCl + Mg \longrightarrow C_2H_5-\overset{\displaystyle C_2H_5}{\underset{\displaystyle C_2H_5}{\overset{|}{\underset{|}{C}}}}OCOCH_3 + MgCl_2 + H_2 \quad \text{(Spassow)}$$

D. RATES OF ALKALINE HYDROLYSIS

The rates of hydrolysis of the acetates with 0.01 N sodium hydroxide, using water or 70% aqueous dioxane as solvents, was followed titrimetically at 20°C. and 30°C.

E. INFRARED SPECTRA

Spectra of all acids, ketones, alcohols and acetates were recorded between 5000 and 625 cm^{-1} with a Baird Infrared Recording Spectrophotometer Model B. All liquids were measured in a sandwich-type sodium chloride cell.

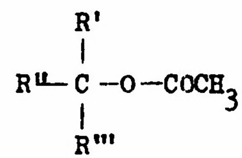
TABLE I. ACETATES OF ALCOHOLS OF FORMULA $R''-C(R')(R''')-CH_2OCOCH_3$

No.	Alcohol	R'	R"	R"'	Carboxylic acid or ester: Method of preparation	Carboxylic acid or ester: Yield in %	Alcohol formation, yield in %	Acetylation of alcohols: Yield in %	Acetylation of alcohols: Method of formation
1.	2-Methyl-1-butanol	CH_3	C_2H_5	H	---	--	--	77	(1)
2.	2,3-Dimethyl-1-butanol	CH_3	$(CH_3)_2CH$	H	A-2	60	90	92	(2)
3.	2,3,3-Trimethyl-1-butanol	CH_3	$(CH_3)_3C$	H	A-6	40	68	89	(1)
4.	2-Ethyl-1-butanol	C_2H_5	C_2H_5	H	---	--	--	70	(1)
5.	3-Methyl-2-ethyl-1-butanol	C_2H_5	$(CH_3)_2CH$	H	A-2	78	83	90	(1)
6.	3,3-Dimethyl-2-ethyl-1-butanol	C_2H_5	$(CH_3)_3C$	H	---	--	96	97	(1)
7.	2-Ethyl-1-hexanol	C_2H_5	$n\text{-}C_4H_9$	H	---	--	--	93	(1)
8.	2-Isopropyl-3-methyl-1-butanol	$(CH_3)_2CH$	$(CH_3)_2CH$	H	A-2 / A-6	50 / 83	87	72	(1)
9.	3,3-Dimethyl-1-butanol	$(CH_3)_3CCH_2$	H	H	A-3		83	81	(2)
10.	Cyclopropanemethanol	CH_2–CH_2–CH (ring)		H	A-5	95	70	89	(1)
11.	Cyclobutanemethanol	CH_2–CH_2–CH_2–CH (ring)		H	---	--	80	87	(1)
12.	Cyclopentanemethanol	----------$(CH_2)_4$--------		H	A-6	65	80	90	(1)
13.	Cyclohexanemethanol	----------$(CH_2)_5$--------		H	---	--	--	77	(1)
14.	Neopentyl alcohol	CH_3	CH_3	CH_3	---	--	91	80	(1)

TABLE I. (Continued)

No.	Alcohol	R'	R"	R"'	Carboxylic acid or ester: Method of preparation	Carboxylic acid or ester: Yield in %	Alcohol formation, yield in %	Acetylation of alcohols: Yield in %	Acetylation of alcohols: Method of formation
15.	2,2-Dimethyl-1-butanol	C_2H_5	CH_3	CH_3	A-1	40	90	92	(1)
16.	2-Methyl-2-ethyl-1-butanol	C_2H_5	C_2H_5	CH_3	A-6	76	83	75	(1)
17.	2,2-Diethyl-1-butanol	C_2H_5	C_2H_5	C_2H_5	A-4	60	65	92	(1)

TABLE II. ACETATES OF SECONDARY AND TERTIARY ALCOHOLS



No.	Compound	R'	R"	R'''	Alcohol formation, yield in %	Acetylation of alcohols: Yield in %	Acetylation of alcohols: Method of formation
18.	Pinacolyl acetate	CH_3	$(CH_3)_3C$	H	87	63 92	(1) (2)
19.	Diisopropylcarbinyl acetate	$(CH_3)_2CH$	$(CH_3)_2CH$	H	93	82 90	(1) (3)
20.	Diisobutylcarbinyl acetate	$(CH_3)_2CHCH_2$	$(CH_3)_2CH{-}CH_2$	H	--	90	(1)
21.	Tertiary-butyl acetate	CH_3	CH_3	CH_3	--	52	(1)
22.	Triethylcarbinyl acetate	C_2H_5	C_2H_5	C_2H_5	--	70	(3)

TABLE III. PHYSICAL PROPERTIES OF CARBOXYLIC ACIDS OR THEIR ESTERS

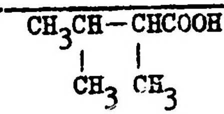
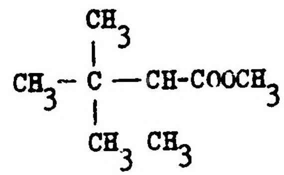
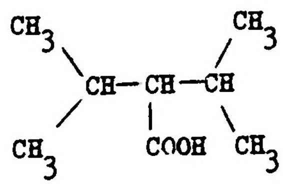
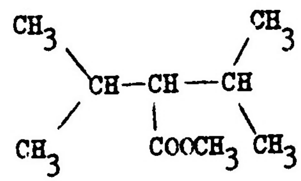
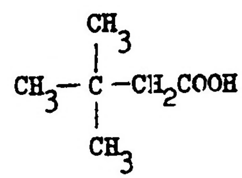
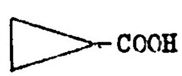
Compound	Structure	Formula	M.P.	B.P. Temp., °C.	B.P. Pressure, mm	Index of refraction	Density
2,3-Dimethylbutyric acid		$C_6H_{12}O_2$	----	188-189	740	n_D^{25} 1.4127	----------
Methyl 2,3,3-Trimethyl Butyrate		$C_8H_{16}O_2$	----	61	30	n_D^{25} 1.4188 n_D^{22} 1.4194	d_{22} 0.8885
Diisopropylacetic acid		$C_8H_{16}O_2$	----	214	740	n_D^{25} 1.4260	d_{25} 0.9125
Methyl diisopropylacetate		$C_9H_{18}O_2$	----	72.5	25	n_D^{20} 1.4159	
3,3-Dimethylbutyric acid		$C_6H_{12}O_2$	5.6	181-181.5	740	n_D^{25} 1.4084	
Cyclopropanecarboxylic acid		$C_4H_6O_2$	----	181-182	745	n_D 1.4363	d_{24} 1.0847

TABLE III. (Continued)

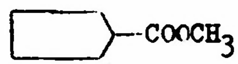
Compound	Structure	Formula	M.P.	B.P. Temp., °C.	B.P. Pressure, mm	Index of refraction	Density
Methyl cyclopentanecarboxylate		$C_7H_{12}O_2$	----	154	740	n_D 1.4353	
2,2-Dimethylbutyric acid	$C_2H_5-C(CH_3)_2-COOH$	$C_6H_{12}O_2$	----	186	745		
Methyl 2,2-Diethylpropionate	$C_2H_5-C(C_2H_5)(CH_3)-COOCH_3$	$C_8H_{16}O_2$	----	62-63	25	n_D^{26} 1.4085	d_{25} 0.8824
Triethylacetic acid	$C_2H_5-C(C_2H_5)_2-COOH$	$C_8H_{16}O_2$	35	131-132	20		
2-Ethyl isovaleric acid	$CH_3-CH(CH_3)-CH(C_2H_5)-COOH$	$C_7H_{14}O_2$	----	196-200	740	n_D 1.4148	

TABLE IV. PHYSICAL PROPERTIES OF BRANCHED PRIMARY AND SECONDARY ALCOHOLS

No.	Compound	Formula	B.P. Temp., °C.	B.P. Pressure, mm	n_D^{25}	d_{25}	$[R_L]_D$ Calcd.	$[R_L]_D$ Found
1.	$C_2H_5-CH(CH_3)-CH_2OH$	$C_5H_{12}O$	128	740	1.4104			
2.	$CH_3CH(CH_3)-CH(CH_3)-CH_2OH$	$C_6H_{14}O$	145-146	741	1.4173			
3.	$CH_3-C(CH_3)(CH_3)-CH(CH_3)-CH_2OH$	$C_7H_{16}O$	158-159	740	1.4230	0.8238	36.05	35.86
4.	$C_2H_5-CH(C_2H_5)CH_2OH$	$C_6H_{14}O$	146-147	741	1.4205			
5.	$CH_3-CH(CH_3)-CH(C_2H_5)-CH_2OH$	$C_7H_{16}O$	84-86	38	1.4234	0.8327	36.05	35.61
6.	$CH_3-C(CH_3)(CH_3)-CH(C_2H_5)-CH_2OH$	$C_8H_{18}O$	88-89	38	1.4348	0.8425	40.66	40.32
7.	$n-C_4H_9-CH(C_2H_5)-CH_2OH$	$C_8H_{18}O$	184	741	1.4280			

TABLE IV. (Continued)

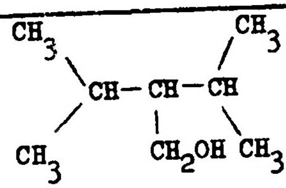
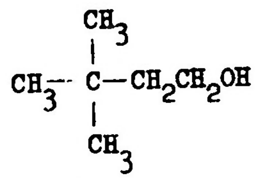
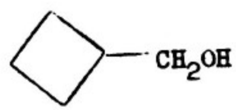
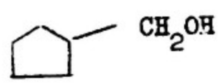
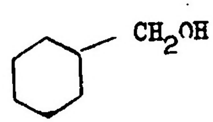
No.	Compound	Formula	B.P. Temp., °C.	B.P. Pressure, mm	n_D^{25}	d_{25}	$[R_L]_D$ Calcd.	$[R_L]_D$ Found
8.		$C_8H_{18}O$	171-172	741	1.4342	0.8425	40.66	40.26
9.		$C_6H_{14}O$	144-145	740	1.4115	0.8097	31.43	31.35
10.		C_4H_8O	123	740	1.4297	0.9098		20.47
11.		$C_5H_{10}O$	140	740	1.4430			
12.		$C_6H_{12}O$	160	740	1.4550			
13.		$C_7H_{14}O$	180	741	1.4634			

TABLE IV. (Continued)

No.	Compound	Formula	B.P. Temp., °C.	B.P. Pressure, mm	n_D^{25}	d_{25}	$[R_L]_D$ Calcd.	$[R_L]_D$ Found
14.	$CH_3-C(CH_3)(CH_3)-CH_2OH$	$C_5H_{12}O$	112-114	740	m.p. 51-51.6°			
15.	$C_2H_5-C(CH_3)(CH_3)-CH_2OH$	$C_6H_{14}O$	134	740	1.4192			
16.	$C_2H_5-C(CH_3)(C_2H_5)-CH_2OH$	$C_7H_{16}O$	152	740	1.4288			
17.	$(C_2H_5)_3C-CH_2OH$	$C_8H_{18}O$	92	25	1.4411			
18.	$CH_3-C(CH_3)(CH_3)-CHOHCH_3$	$C_6H_{14}O$	120-120.5	741	1.4153	0.8122	31.43	31.50
19.	$(CH_3)_2CH-CHOHCH(CH_3)-CH_3$	$C_7H_{16}O$	139-139.5	740	1.4210	0.8245	36.05	35.71

TABLE IV. (Continued)

No.	Compound	Formula	B.P. Temp., °C.	B.P. Pressure, mm	n_D^{25}	d_{25}	$[R_L]_D$ Calcd.	$[R_L]_D$ Found
20.	$(CH_3)_2CHCH_2CHOHCH_2CH(CH_3)_2$	$C_9H_{20}O$						
21.	$(C_2H_5)_3COH$	$C_7H_{16}O$	140-141	740	1.4256			

TABLE V. PHYSICAL PROPERTIES OF ACETATES OF HIGHLY-BRANCHED ALCOHOLS

No.	Acetate of:	Formula	B.P.		n_D^{25}	d_{25}	$[R_L]_D$	
			°C	Pressure, mm			Calcd.	Found
1.	2-Methyl-1-butanol	$C_7H_{14}O_2$	138-139	741	1.3996	0.8719	36.17	36.17
2.	2,3-Dimethyl-1-butanol	$C_8H_{16}O_2$	147	740	1.4068	0.8790	40.79	40.50
3.	2,3,3-Trimethyl-1-butanol	$C_9H_{18}O_2$	170-171	741	1.4125	0.8687	45.41	45.30
4.	2-Ethyl-1-butanol	$C_8H_{16}O_2$	160	740	1.4090	0.8764	40.79	40.68
5.	3-Methyl-2-ethyl-1-butanol	$C_9H_{18}O_2$	88-89	30	1.4156	0.8774	45.41	45.23
6.	3,3-Dimethyl-2-ethyl-1-butanol	$C_{10}H_{20}O_2$	100	40	1.4220	0.8770	50.0	49.90
7.	2-Ethyl-1-hexanol	$C_{10}H_{20}O_2$	104	40	1.4182	0.8688	50.0	49.97
8.	3,3-Dimethyl-1-butanol	$C_8H_{16}O_2$	156-157	741	1.4038	0.8683	40.79	40.60
9.	2-Isopropyl-3-methyl-1-butanol	$C_{10}H_{20}O_2$	130	110	1.4200	0.8803	50.0	49.5
10.	Cyclopropanemethanol	$C_6H_{10}O_2$	133.5	741	1.4156	0.9603		
11.	Cyclobutanemethanol	$C_7H_{12}O_2$	150	741	1.4245	0.9508		
12.	Cyclopentanemethanol	$C_8H_{14}O_2$	172.5	741	1.4340	0.9577		
13.	Cyclohexanemethanol	$C_9H_{16}O_2$	108	40	1.4422	0.9541		
14.	Neopentyl alcohol	$C_7H_{14}O_2$	127	740	1.3927	0.8539	36.17	36.36
15.	2,2-Dimethyl-1-butanol	$C_8H_{16}O_2$	152-153	740	1.4050	0.8704	40.79	40.60
16.	2-Methyl-2-ethyl-1-butanol	$C_9H_{18}O_2$	100	80	1.4150	0.8815	45.41	44.95

TABLE V. (Continued)

No.	Acetate of:	Formula	B.P. °C	B.P. Pressure, mm	n_D^{25}	d_{25}	$[R_L]_D$ Calcd.	$[R_L]_D$ Found
17.	2,2-Diethyl-1-butanol	$C_{10}H_{20}O_2$	103-104	40	1.4269	0.8900	50.00	49.68
18.	Pinacolyc alcohol	$C_8H_{16}O_2$	138-138.5	738	1.4002			
19.	Diisopropylcarbinol	$C_9H_{18}O_2$	161	745	1.4110*			
20.	Diisobutylcarbinol	$C_{11}H_{22}O_2$	125-126	60	1.4117			
21.	Trimethylcarbinol	$C_6H_{12}O_2$	96.5-97.0	745	1.3840			
22.	Triethylcarbinol	$C_9H_{18}O_2$	163	740	1.4270			

* n_D^{20}

TABLE VI. RATE OF SAPONIFICATION OF ACETATES OF PRIMARY ALCOHOLS

No.	Compound	Solvent	Normality of NaOH	Molarity of ester	Rate constant, k_2, g. mol. l./min⁻¹ 20°C.	Rate constant, k_2, g. mol. l./min⁻¹ 30°C.	$\frac{k_{ethyl\ acetate}}{k_{alkyl\ acetate}}$
		Water	0.012	0.0099	1.420	----	
1.	2-Methyl-1-butyl acetate			0.0075			
		70% Dioxane	0.015	0.008	0.47	----	3.0
			0.0119	0.009	----	0.78	
2.	2,3-Dimethyl-1-butyl acetate	70% Dioxane		0.009	0.35	----	
			0.0133	0.008			3.3
			0.0119	0.009	----	0.72	
3.	2,3,3-Trimethyl-1-butyl acetate	70% Dioxane	0.0123	0.007	0.324	----	3.7
				0.009			
4.	2-Ethyl-1-butyl acetate	70% Dioxane	0.0133	0.0109	0.24	----	
				0.0090			4.8
			0.0119	0.0093	----	0.49	
				0.0100			
5.	3-Methyl-2-ethyl-1-butyl acetate	70% Dioxane	0.015	0.010	0.23	----	
				0.008			5.8
			0.0119	0.009	----	0.41	
				0.010			
6.	3,3-Dimethyl-2-ethyl-1-butyl acetate	70% Dioxane	0.015	0.0107	0.064	----	
				0.0093			21.0
			0.0119	0.0095	-----	0.11	
				0.0100			
7.	2-Ethyl-1-hexyl acetate	70% Dioxane	0.0125	0.0098	0.156	----	
			0.0119	0.0102	-----	0.36	6.5
				0.0085			
8.	3,3-Dimethyl-1-butyl acetate	70% Dioxane	0.0123	0.0105	0.627	----	1.9
				0.0087			

TABLE VI. (Continued)

No.	Compound	Solvent	Normality of NaOH	Molarity of ester	Rate constant, k_2, g. mol. l./min^{-1} 20°C.	Rate constant, k_2, g. mol. l./min^{-1} 30°C.	$\frac{k_{ethyl\ acetate}}{k_{alkyl\ acetate}}$
9.	2-Isopropyl-3-methyl-1-butyl acetate	70% Dioxane	0.0122	0.0087 0.0078	0.092	----	13.3
10.	Cyclopropanemethyl acetate	70% Dioxane	0.0133	0.0084 0.0100	1.08	----	1.004
			0.0119	0.0086 0.0084	----	2.28	
11.	Cyclobutanemethyl acetate	70% Dioxane	0.0133	0.0084 0.0096	0.74	----	1.6
			0.0119	0.0090 0.0080	----	1.47	
12.	Cyclopentanemethyl acetate	70% Dioxane	0.0133	0.0093 0.0087 0.0080	0.54	----	2.2
			0.0119	0.0096 0.0081	----	1.08	
13.	Cyclohexanemethyl acetate	70% Dioxane	0.0135	0.0092 0.0100	0.33	----	3.4
			0.0119	0.0101 0.0098	----	0.70	
14.	Neopentyl acetate	Water	0.0122	0.0074 0.0069	0.86	----	4.8
		70% Dioxane	0.015	0.0078	0.28		
			0.0119	0.0083 0.0100	----	0.50	

TABLE VI. (Continued)

No.	Compound	Solvent	Normality of NaOH	Molarity of ester	Rate constant, k_2, g. mol. l./min^{-1} 20°C.	30°C.	$k_{ethyl\ acetate}$ / $k_{alkyl\ acetate}$
15.	2,2-Dimethyl-1-butyl acetate	Water	0.012	0.007	0.70	----	
		70% Dioxane	0.015	0.0101 0.0088	0.21	----	7.0
			0.0119	0.0090 0.0082	----	0.34	
16.	2-Methyl-2-ethyl-1-butyl acetate	70% Dioxane	0.0123	0.0087 0.0090	0.124	----	12.0
17.	2,2-Diethyl-1-butyl acetate	70% Dioxane	0.0133	0.0092 0.0069	0.050	----	22.5
			0.0119	0.0093 0.0080	-----	0.108	

TABLE VII. RATE OF SAPONIFICATION OF ACETATES OF SECONDARY ALCOHOLS

No.	Compound	Solvent	Normality of NaOH	Molarity of ester	Rate constant, k_2, g. mol. l./min^{-1} 20°C.	30°C.	$\frac{k_{\text{isopropyl acetate}}}{k_{\text{alkyl acetate}}}$
1.	Pinacolyl acetate	70% Dioxane	0.0245	0.0100 0.0100	0.035	----	11.3
			0.012	0.0108	-----	0.054	
2.	Diisobutylcarbinyl acetate	70% Dioxane	0.025	0.0079 0.0091	-----	0.0236	25.0
3.	Tert. butyl acetate	70% Dioxane	0.025	0.0075 0.0090	-----	0.0381	----
4.	Diisopropylcarbinyl acetate(*)	70% Dioxane	0.025	0.0079	-----	0.0109	56.0

(*) Rate constant calculated graphically by extrapolating the line obtained from 30% to 80% hydrolysis.

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to

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RF Project 497

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Investigator S. Sarel Date 8-10-54

Supervisor Melvin S. Newman Date 8-10-54

For The Ohio State University Research Foundation

Executive Director Orvin C. Woolpert Date 8/10/54

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